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UREA GREASE COMPOSITION
[Ureakei gurisu soseibutsu]

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[Claim(s)]

[Claim 1] Urea grease composition prepared by mixing the base oil consisting of mineral oil and/or synthetic oil with the thickener in an amount of 2 - 20 wt.%, wherein the thickener consists of a mixture of diurea compound expressed as the formula (a) and formula (b) described below at a molar ratio of the compound (b) to the compound (a) of 20 - 90 mol%:

(a) $R^1NHCONHR^2NHCONHR^3$ (wherein R^2 is a tolylene group; and R^1 and R^3 are each a linear or branched saturated or unsaturated hydrocarbon group having 16 - 18 carbons),

(b) $R^4NHCONHR^5NHCONHR^6$ (wherein R^5 is a diphenylmethane group; and R^4 and R^6 are each an linear or branched saturated hydrocarbon group having 8 carbons)

[Detailed Explanation of this Invention]

[0001] [Industrial Application]

This invention relates to the urea grease composition prepared by combining two kinds of diurea compounds as a thickening agent.

[0002] [Description of the Prior Art]

In recent years, in various industries, such as automobile, steel, etc., remarkable advancements has been made to the machinery techniques, allowing various machine parts to be smaller, lighter, and higher in capacity. Thereby, the temperature of lubrication parts tends to be higher. Thus, the application of a urea type grease having excellent thermal resistance or oxidation stability,

effective for reduction of usage amount and machine life extension, has gradually increased. The technique disclosed in Pat. No. 1-284591,A reported the diurea compound as a thickening agent. This diurea compound has an alkyl group carrying 3 - 4 carbons at one side, and an alkyl group carrying 16 - 18 carbons at another side of a divalent diphenyl methane radial. Although the technique of Pat. No. 1-268793,A also uses the diurea compound as a thickening agent, a radical selected from a group consisting of octadecyl radical, octyl radical, and dodecyl radical is connected to both ends of divalent diphenyl methane radical of this diurea compound. Moreover, the mol ratio of the octadecyl radical is arranged as 20 - 80 mol%. Pat. No. 1-139696,A is related to a technique of producing a thickener by combining a diurea compound having a urea bond at both terminals of diphenylmethane and a diurea compound having a urea bond at both terminals of tolylene group or bitolylene group. However, alkyl connected to diphenylmethane group through a urea bond is limited to the alky having 8 carbons. Also, a material connected to a tolylene group or bitolylene group through a urea bond is limited to an aromatic group. Moreover, Pat. No. 2-77494,A disclosed to a technique of producing a thickener by combining a diurea compound having a urea bond at both ends of diphenyl methane and diurea compound having a urea bond at both terminals of bitolylene. Although the technique described in Pat. No. 63-26798,B uses three kinds of diurea compounds, the same material is used as every group

placed between two urea bonds. In addition, this method combines a compound having a dodecyl at both ends, a compound having dodecyl at one end and octyl at the other end, and a compound having octyl at both ends. Although the method disclosed in Pat. No. 58-185693,A adds alkenyl amber acid amide, alkylbenzene-sulfonic acid metal salt, petroleum sulfonic acid metal salt, etc. in a diurea type grease so as to improve the acoustic feature, there is no particular characteristic in the diurea.

[0003] [Objects of the Invention]

The first purpose of this invention is to provide urea grease which can provide desired thickening with a small amount of use, excellent mechanical stability, water resistance, pumpability, and acoustic property. The second object of this invention is to expand and improve the technique disclosed in Pat. No. 1-1396.

[0004] [Elements of the Invention]

This invention relates to a urea grease composition prepared by mixing the base oil consisting of mineral oil and/or synthetic oil with the thickener in an amount of 2 - 20 wt.%, wherein the thickener consists of a mixture of diurea compound expressed as the formula (a) and formula (b) described below at a molar ratio of the compound (b) to the compound (a) of 20 - 90 mol%:

(a) $R^1NHCONHR^2NHCONHR^3$ (wherein R^2 is a tolylene group; and R^1 and R^3 are each a linear or branched saturated or unsaturated hydrocarbon group having 16 - 18 carbons),

(b) $R^4NHCONHR^5NHCONHR^6$ (wherein R^5 is a diphenylmethane group; and R^4 and R^6 are each a linear or branched saturated hydrocarbon group having 8 carbons).

[0005] The component (a) can be obtained by reacting tolylene diisocyanate, hexadecyl amine, heptadecyl amine, and/or an octadecyl amine. Tolylene diisocyanate can be 2,4-tolylene diisocyanate, 2,6-tolylene diisocyanate, their mixture, etc.

The component (b) can be obtained by reacting diphenylmethane diisocyanate and octyl amine.

[0006] [Operational Examples]

Hereafter, this invention is explained by referring to operational examples. Moreover, comparison examples are explained for clarifying the characteristic of the urea grease of this invention.

Operational examples 1 - 7:

After putting diisocyanate as the component (a) at a ratio described in the table and the base oil of 60 wt sections into a grease iron pot, the mixture was heated to about 80°C to dissolve the diisocyanate. Then, after n-octadecyl amine as the component (a) dissolved in 20 wt parts of base oil was gradually added, the mixture was agitated vigorously. After about 10 minutes, diphenylmethane-4,4'-diisocyanate as the component (b) was added. Then, after n-octyl amine dissolved in 20 wt parts of base oil was added, the mixture was continuously stirred. Although the temperature increased

by the reaction of diisocyanate and amine, the mixture was kept stirred in this condition for about 30 minutes. Then, the mixture was heated to 170°C to complete the reaction. Then, the reacted product was cooled naturally and kneaded to the room temperature to prepare grease. The viscosity of the mineral oil shown in the operational example was 11 cst (100°C), and the poly α olefin oil was 12 cst (100°C). The table shows the thickening consistency, dropping point, shell roll (150°C, 24 h), and the thickening consistency after heating at 180°C (25°C, non-mixing), and the result of acoustic test of each operational example. Note that the acoustic test was measured based on the method described in Pat. No. 53-2357,B.

Comparison examples 1 - 2:

Diisocyanate at a ratio described in the table and 80 wt parts of mineral oil were put into the grease iron pot, heated to about 80°C to dissolve diisocyanate, to which amine dissolved in 20 wt parts of mineral oil was added. Then, the mixture was stirred. The mixture was stirred in this condition for about 30 min., heated to about 170°C to complete the reaction, cooled to room temperature naturally, and kneaded to form grease. The property of each comparison example is shown in the table.

[0007] [Table 1]

		実施例1	実施例2	実施例3	実施例4	実施例5
成分(a)	(1)2.4/2.6 (80%/20%)－トリレンジイソシアネートg	3.22	2.82	2.02	1.21	0.40
	(2)n－オクタデシルアミンg	9.58	8.38	5.98	3.59	1.20
成分(b)	(1)ジフェニルメタン4,4′－ジイソシアネート g	1.57	2.36	3.94	5.51	7.08
	(2)n－オクチルアミンC8g	1.63	2.44	4.06	5.69	7.32
鉱油 g		184	184	184	184	184
ポリα－オレフイン g		－	－	－	－	－
増ちょう剤含有量 %		8.0	8.0	8.0	8.0	8.0
(a)/(b) モル%／モル%		80/20	70/30	50/50	30/70	10/90
(性状)						
ちょう度 (25℃、混和)		268	255	240	245	271
シェルロール (室温，24時間)		358	347	317	298	332
含水10%シェルロール (室温，24時間)		362	356	347	309	351
シェルロール (150℃，24時間)		382	369	310	305	321
音響試験 (120秒後)		38	20	25	15	26

Key for Table 1:

		Operational example 1	Operational example 2	Operational example 3	Operational example 4	Operational example 5
Component (a)	(1) 2.4/2.6 (80%/20%)-tolylene di isocyanate g; (2) n-octadecyl amine g					
Component (b)	(1) diphenyl methane 4,4-di isocyanate g; (2) n-octyl amine C 8g					
Mineral oil						
Poly α olefin						
Thickener content (a)/(b) mol%/mol% (Condition)						
Consistency (25°C, Mixed)						
Shell roll (room temp. 24 h)						
Shell roll with 10% water (room temp. 24 h)						
Shell roll (150°C, 24 h) Acoustic test (After 120 sec.)						

[0008] [Table 2]

		実施例 6	実施例 7	比較例 1	比較例 2
成分 (a)	(1)2.4/2.6 (80%／20%) －トリレンジイソシアネートg	0.91	1.21	* 7.56	－
	(2)n－オクタデシルアミンg	2.69	3.59	22.44	－
成分 (b)	(1)ジフェニルメタン4,4′－ジイソシアネート g	4.13	5.51	－	7.87
	(2)n－オクチルアミンC 8g	4.27	5.69	－	8.13
鉱油 g		188	－	170	184
ポリα－オレフイン g		－	184	－	－
増ちょう剤含有量 %		6.0	8.0	15.0	8.0
(a)/(b) モル%／モル%		30/70	30/70	100/0	0/100
(性状)					
ちょう度 (25℃、混和)		292	251	343	342
シェルロール (室温, 24時間)		357	313	368	402
含水10%シェルロール (室温, 24時間)		382	316	331	412
シェルロール (150℃, 24時間)		362	314	>440	365
音響試験 (120秒後)		20	18	47	51

*The ratio of 2.4/2.6 in the comparison example is 65%/35%.
Thickening consistency: JIS 2220
Shell roll: According to ASTM D1831
Acoustic test: According to the method described in Pat. No. 53-2357,B.

Key for Table 2:

		Operational example 6	Operational example 7	Comparison example 1	Comparison example 2
Component (a)	(1) 2.4/2.6 (80%/20%)-tolylene di isocyanate g; (2) n-octadecyl amine g				
Component (b)	(1) diphenyl methane 4,4-di isocyanate g; (2) n-octyl amine C 8g				
Mineral oil Poly α olefin Thickener content (a)/(b) mol%/mol% (Condition) Consistency (25°C, Mixed) Shell roll (room temp. 24 h)					
Shell roll with 10% water (room temp. 24 h)					
Shell roll (150°C, 24 h) Acoustic test (After 120 sec.)					

[0009] Operational example 8, comparison examples 3 - 5:

As the operational example 8, grease was prepared by adding the antioxidant, anti-rust agent, and extreme pressure agent to the operational example 6. The characteristic of this grease was compared with the grease available on the market. The results are shown in the table below. The grease of this invention can provide superior mechanical stability, water resistance, thermal resistance, and pumpability.

[Table 3]

	実施例 8	比較例 3	比較例 4	比較例 5
(性　　状)				
ちょう度　(25℃、混和)	313	272	320	338
シェルロール　(室温、24時間)	360	414	365	>440
含水10%シェルロール (室温、24時間)	391	>440	>440	>440
シェルロール　(150℃、24時間)	372	>440	>440	>440
見掛粘度　0℃、10 sec^{-1} (ポアズ)	910	−	1180	1800

Key:

	Operational example 8	Comparison example 3	Comparison example 4	Comparison example 5
(Characteristic) Thickening consistency (25°C, Mixed)				
Shell roll (room temp. 24 h)				
Shell roll with 10% water (room temp. 24 h)				
Shell roll (150°C, 24h)				
Apparent viscosity (poise)				

(Note)
Comparison example 3: Urea type grease on the market.
Comparison example 4: Lithium soap grease on the market.
Comparison example 5: Calcium complex type grease

[0010] Comparison examples 6 - 10:

The data on the Operational examples 1 - 4 and 9 described in Pat. No. 1-139696,A are shown as Comparison examples 6 - 10.

[Table 4]

	比較例	6	7	8	9	10
	特開平1－139696号 発明の対応実施例番号	1	2	3	4	9
成分(a)	(1)ジフェニルメタン4, 4′－ジイソシアネート g	8.98	6.08	3.09	6.69	8.98
	(2)オクチルアミンC8 g	9.29	6.29	3.19	6.92	9.29
成分(b)	(3)3,3′-ビトリレン-4,4′-ジイソシアネート g	3.16	6.42	9.79		3.16
	(4)2.4/2.6 (65%/35%) －トリレンジイソシアネート g				4.66	
	(5)パラトルイジン g	2.57	5.21	7.93	5.73	2.57
	鉱油 g	176	176	176	176	
	ポリα－オレフィン g					176
	増ちょう剤含有量 %	12	12	12	12	12
	(a)/(b) モル%／モル%	75/25	50/50	25/75	50/50	50/50
性状	ちょう度 (25℃、混和)	270	285	295	290	283
	シェルロール (150℃, 24時間)	283	296	325	341	291
	150℃加熱後ちょう度 (25℃、不混和)	178	175	174	177	179
	音響試験 (120秒後)	52	59	67	48	49

Key for Table 4:

Comparison example		6	7	8	9	10
Pat. No. 1-139696 Corresponding operational example						
Component (a)	(1) Diphenyl methane 4,4′ di isocyanate g; (2) Octyl amine C8 g					
Component (b)	(3) 3,3′-bitrylene-4,4′-di isocyanate g; (4) 2.4/2.6 (65%/35%) - tolylene di isocyanate g; (5) Paratolydine g					
Mineral oil Poly α olefin Thickener content (a)/(b) mol%/ mol%						
Characteristic	Thickening consistency (25°C, Mixed) Shell roll (room temp. 24 h)					
Thickening consistency after heated at 150°C (25°C, non-mixed) Acoustic test (After 120 sec.)						

As shown in the table, compared with the invention disclosed in Pat. No. 1-139696, this invention can provide improved overall

acoustic effect. Furthermore, with the method based on this invention, harder grease can be obtained from a smaller amount of thickener. In other words, this invention can clearly provide improved thickening yield. Thereby, the urea type grease composition of this invention not only improves the acoustic property, but also provides excellent thickening yield with water resistance and mechanical stability from room temperature to high temperature.

[0011] [Effectiveness]

The new thickener composition provided by this invention can enrich the technique and improve the main problem of the acoustic characteristic of the prior technology disclosed in Pat. No. 1-139696. Furthermore, the grease of this invention provides excellent mechanical stability at high temperature and is extremely low in thermosetting.